Portable Parallel Beam X-Ray Diffraction System for In-Line Process Control in the Steel Industry

Ian Radley : Director of Advanced Product Development

Project Overview

- **Project description**
  - Design, build and test an on-line sensor for zeta phase measurement on a steel galvannealing line.

- **Objectives**
  - Prove the use of parallel beam x-ray diffraction in an industrial environment
  - Demonstrate the capability of low power x-ray systems to replicate or exceed conventional laboratory instruments through the application of x-ray optics

    As the importance of using existing work became obvious in this project a new objective emerged
  - Show the potential of early prototype modular systems to address multiple sensor applications

- **Overall goal**
  - Demonstrate energy saving through the on-line application of x-ray optic based sensors.

Technical Merit

- **Contributes new information or technology to the S/C community**
  - X-ray techniques provide measurement of crystalline properties and composition.
  - X-ray optic based sensors represent a critical, significant advancement over current S/C capabilities. X-ray applications are currently restricted mainly to laboratory applications.
  - X-ray optics relaxes both the geometric requirements and the power requirements through optic processing of the x-ray beam.
  - On-line measurement capabilities exceeding those conventionally available in the laboratory are now possible.

Technical Merit

- X-ray optics are an enabling technology that make diffraction based sensors viable.
  - Allow for low power sources to be used without sacrificing count rate
  - Insensitive to sample position – relaxes geometric requirements
  - Allow for no moving system parts
  - Insensitive to environmental changes

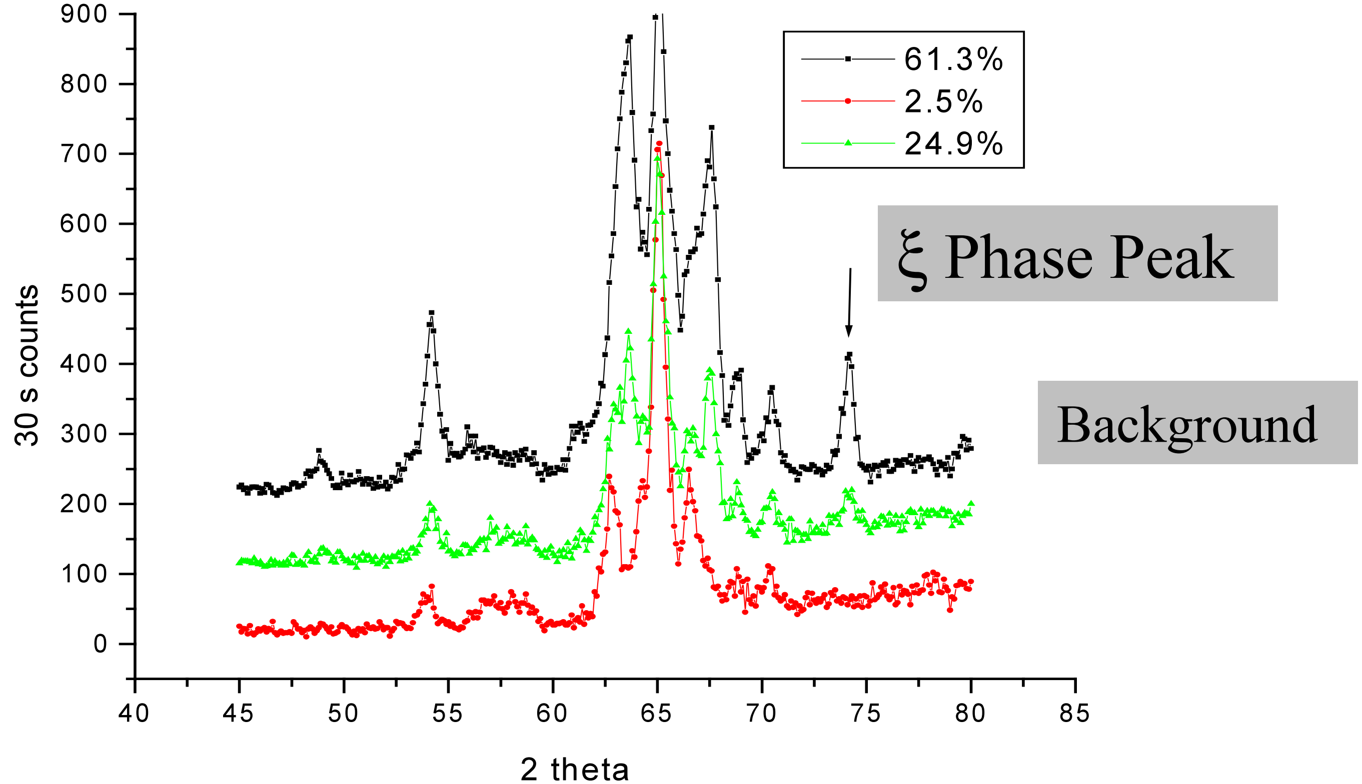
Technical Progress and Outlook

Milestone	Due	Actual	Comments (includes 6 m extension to co-ordinate testing at AK Steel)
Proof of concept	7/00	7/00	Phase 1 SBIR
Sensor component selection	12/01	12/01	Completed evaluation and selection of sources, detectors and filters
Bench top testing	2/02	2/02	Assembly of selected components in bench-top sensor simulation
Sensor assembly	7/02	8/02	Assembly of electronics and mechanical components with x-ray system
Sensor pretest at XOS	8/02	9/02	Simulation of on-line installation with fixed steel plates
Installation at AK Steel	9/02	10/02	Installation on galvanneal line
On line data collection	10/02	10/02	Data collection over a series of galvanneal runs
Final report	12/02	3/03	Report submission

Flexible lab based systems allowed for the optimization of the sensor parameters.

Galvanneal Spectrum

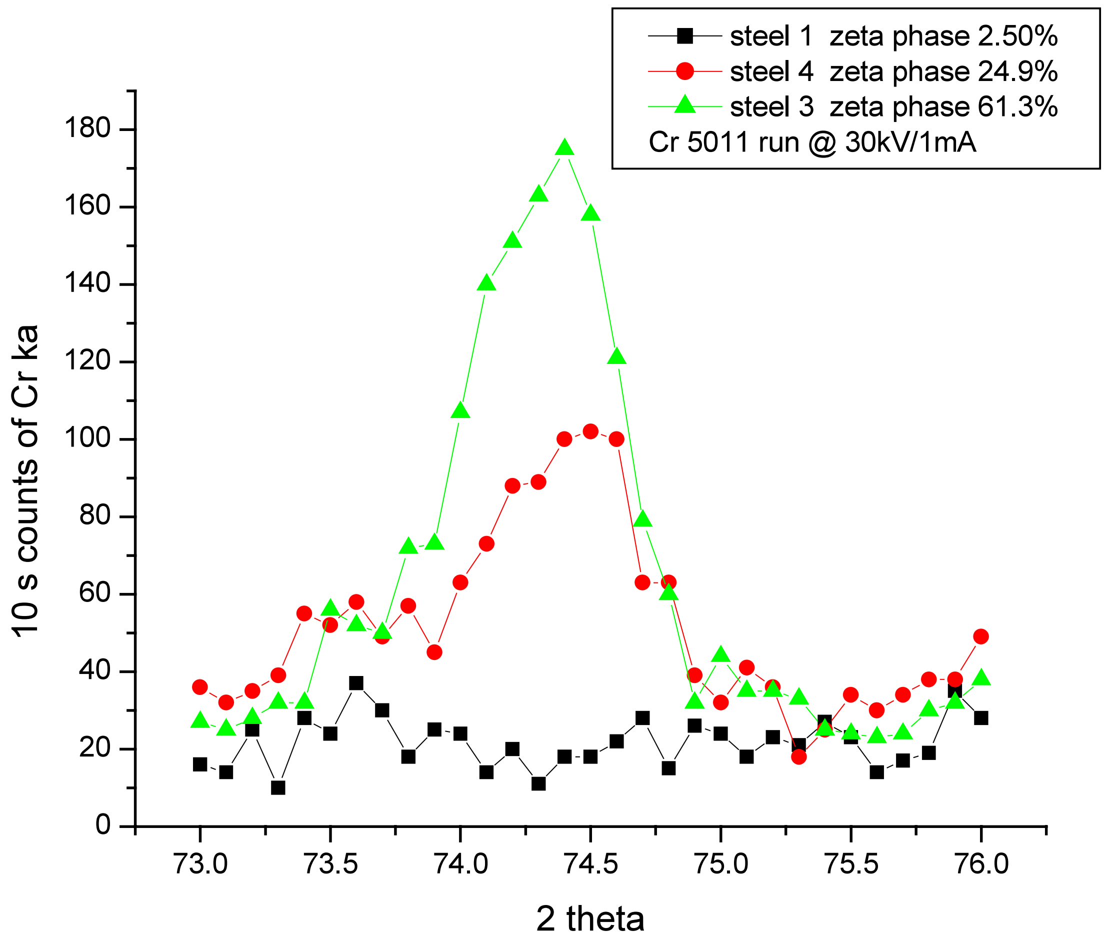
The Red curve is the baseline. Other curves have been shifted 100 counts each for clarity



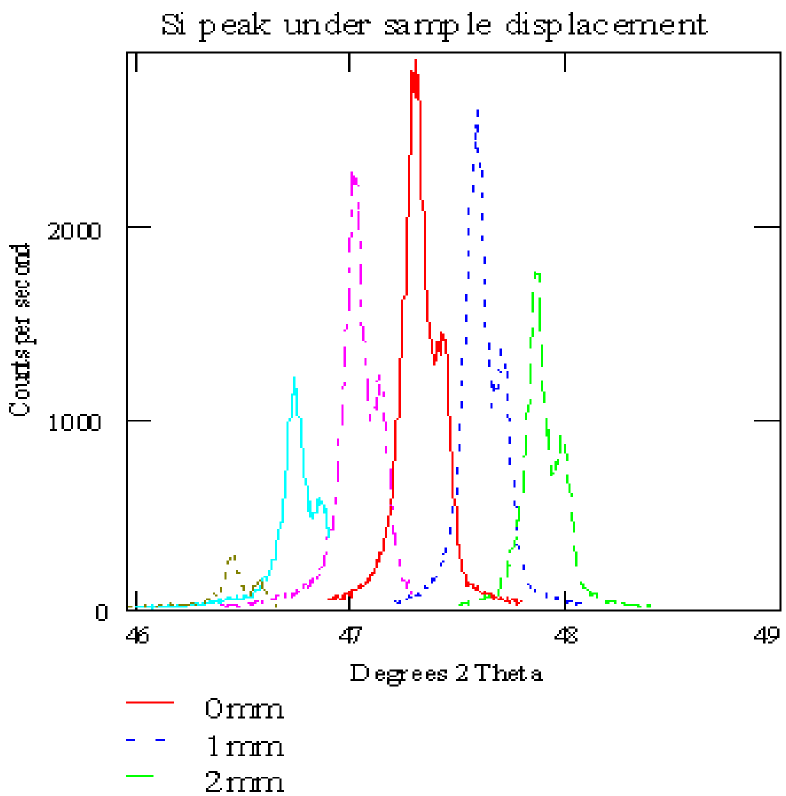
This data achieved with Cu Kα radiation at 8.04KeV with a cooled PIN diode detector. Energy resolution is required due to interference from the Zn line at 8.67 KeV

Energy spectra can be chosen so that background noise is reduce and signal to noise ratio's are improved.

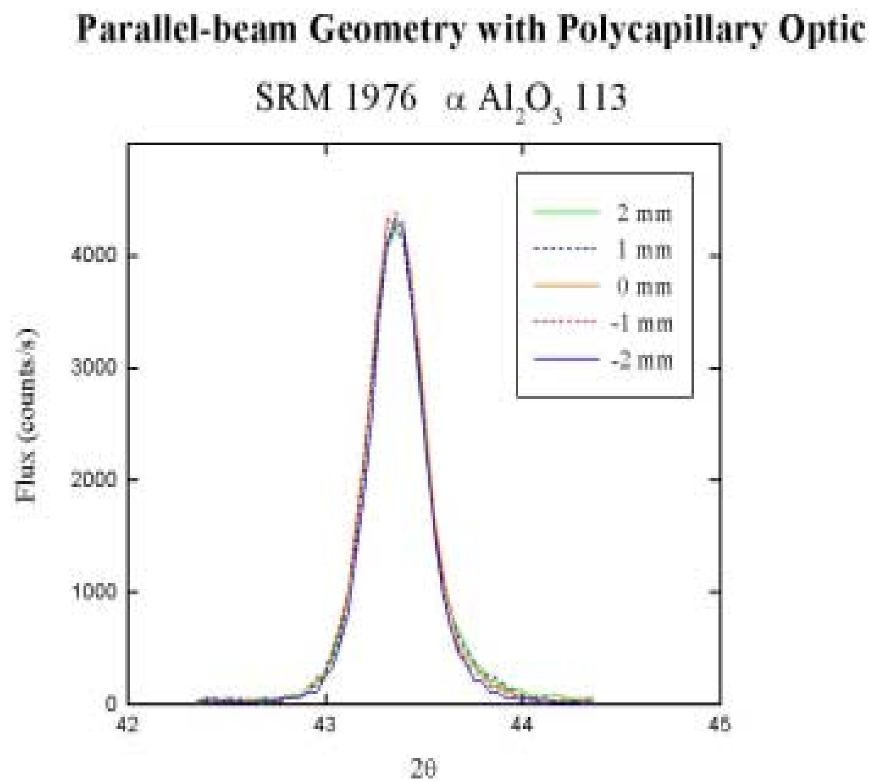
Data achieved with Cr Kα at 6.67 KeV with no adjacent fluorescent lines. This relaxes the detector resolution requirement and allows the use of proportional counters



Technical Merit – Optics make sensors practical for on-line applications.

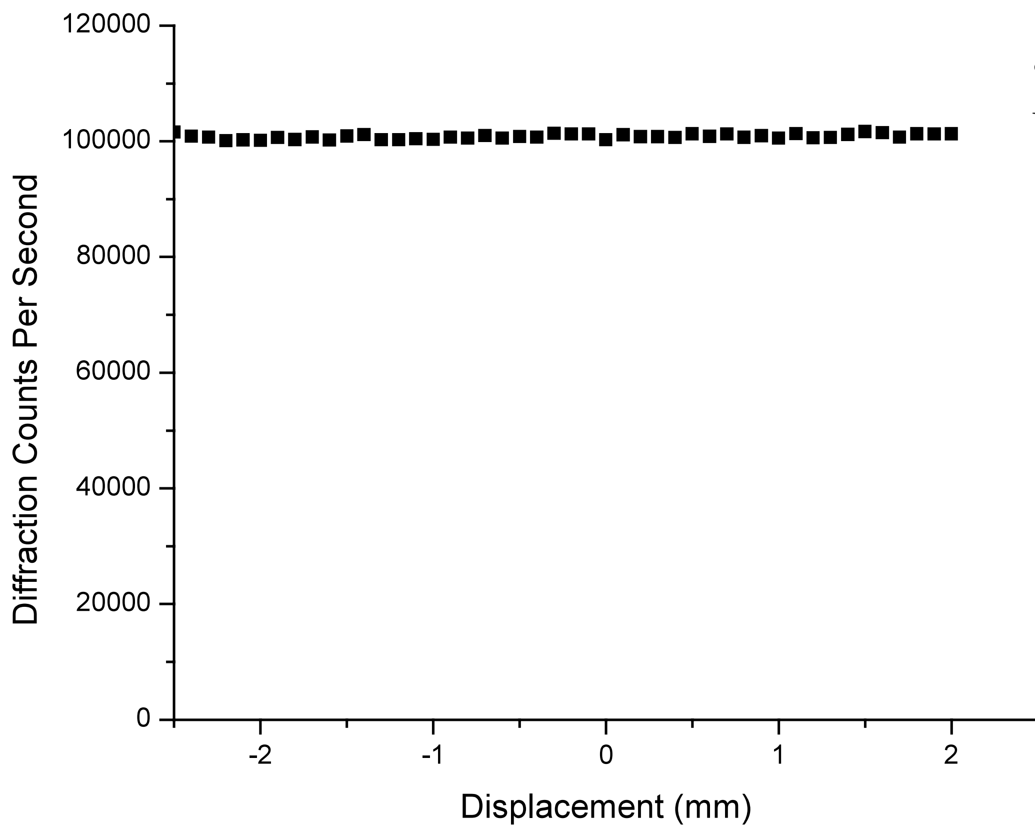


Para-focusing Geometry



Parallel Beam Geometry

Technical Merit – parallel beam geometry significantly reduces the sensitivity to sample displacement.

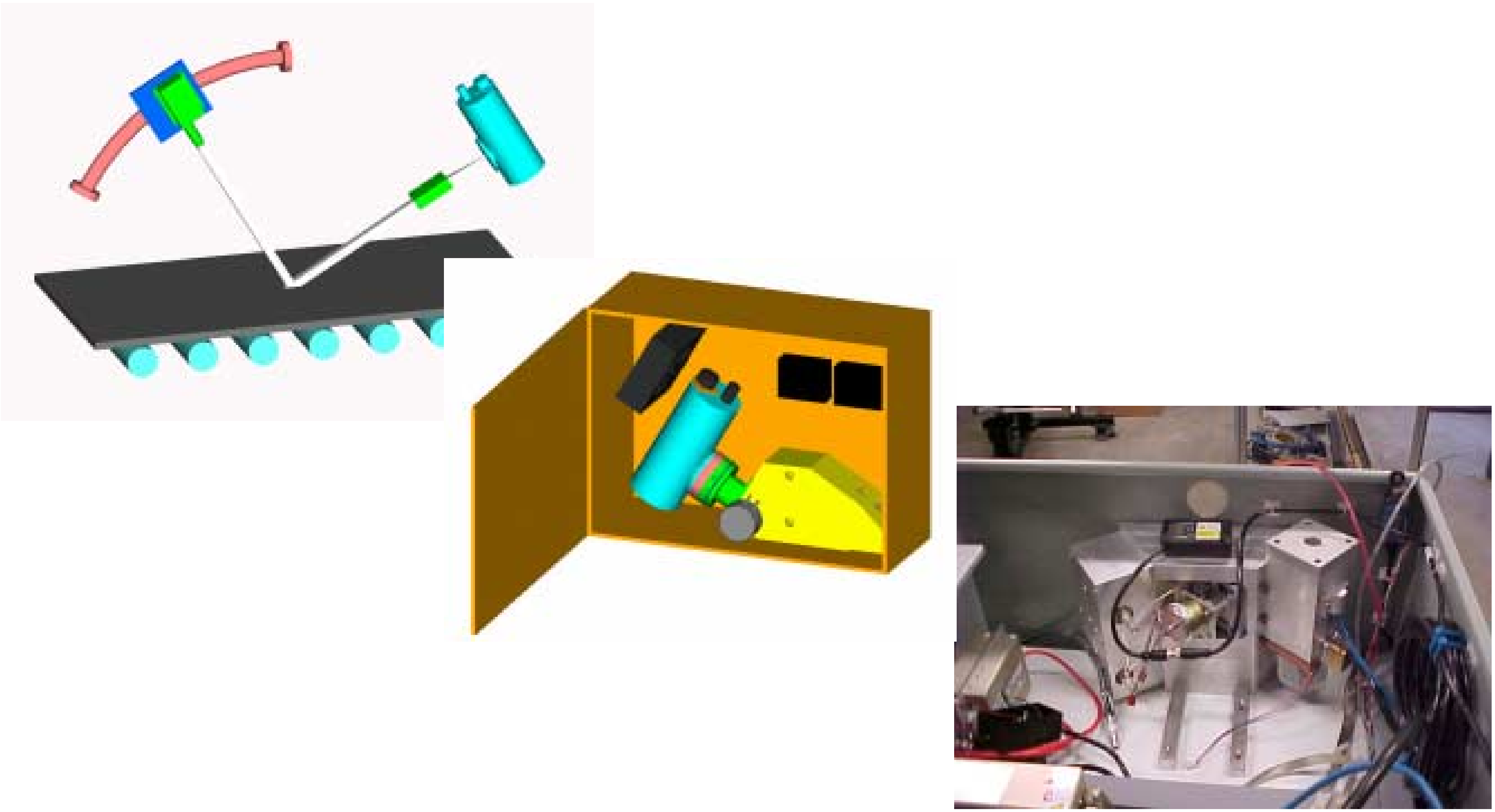


This insensitivity allows the use of unprepared samples

- Curved
- Rough
- Vibrating

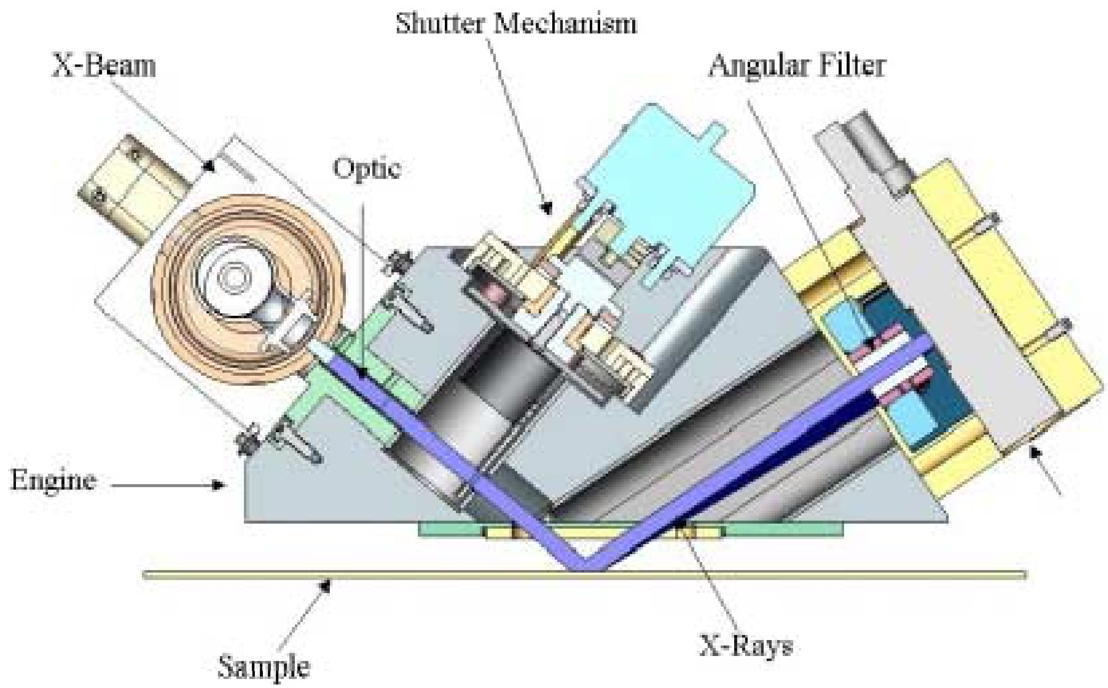
Diffraction peak integrated intensity as a function of sample displacement at a fixed diffraction angle.

Evolution from concept to product



Evolution from original solid model to (nearly) final product

Diffraction engine detail



Section of diffraction engine

Control cabinet

Control cabinet based on PLC technology

PLC technology provides robust industrial control and factory data integration capability

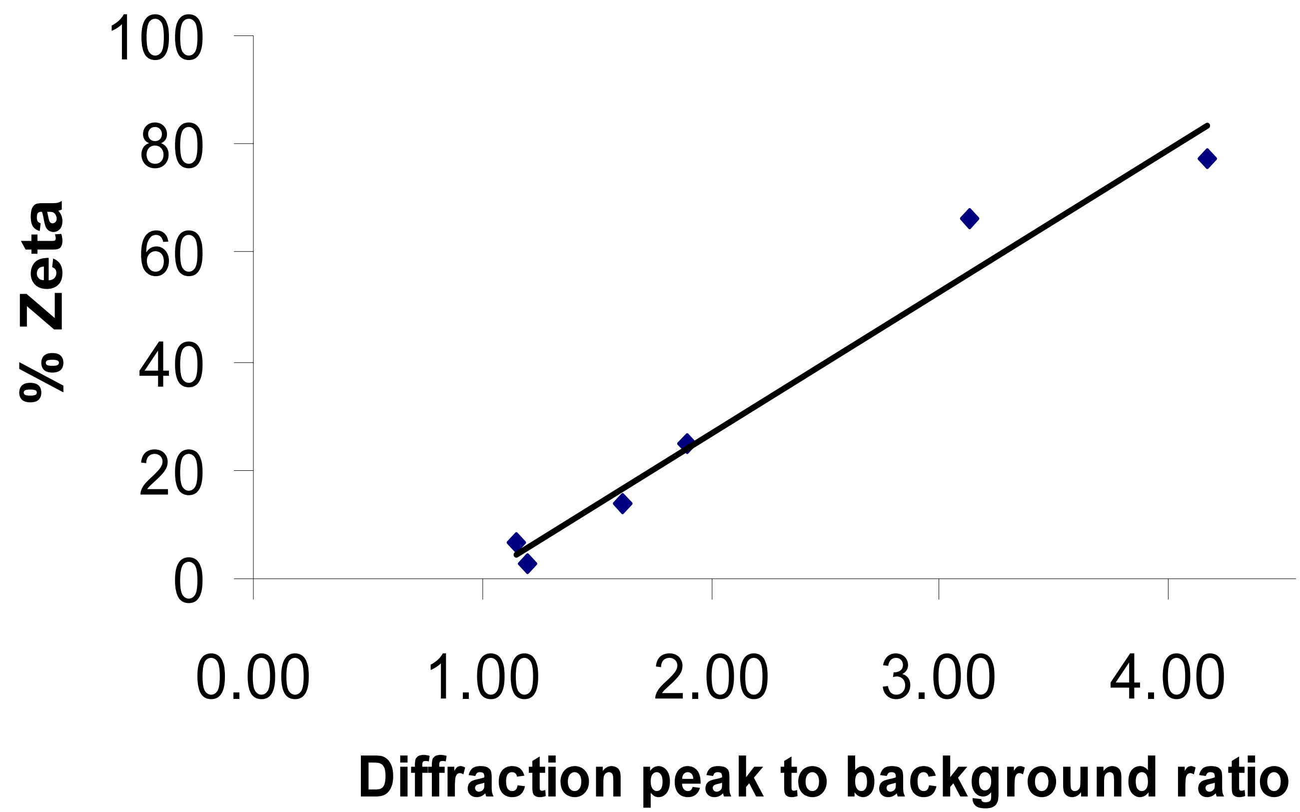
Beta unit at AK Steel

Finished unit showing control cabinet and engine cabinet

Installation at a galvanneal steel production line.

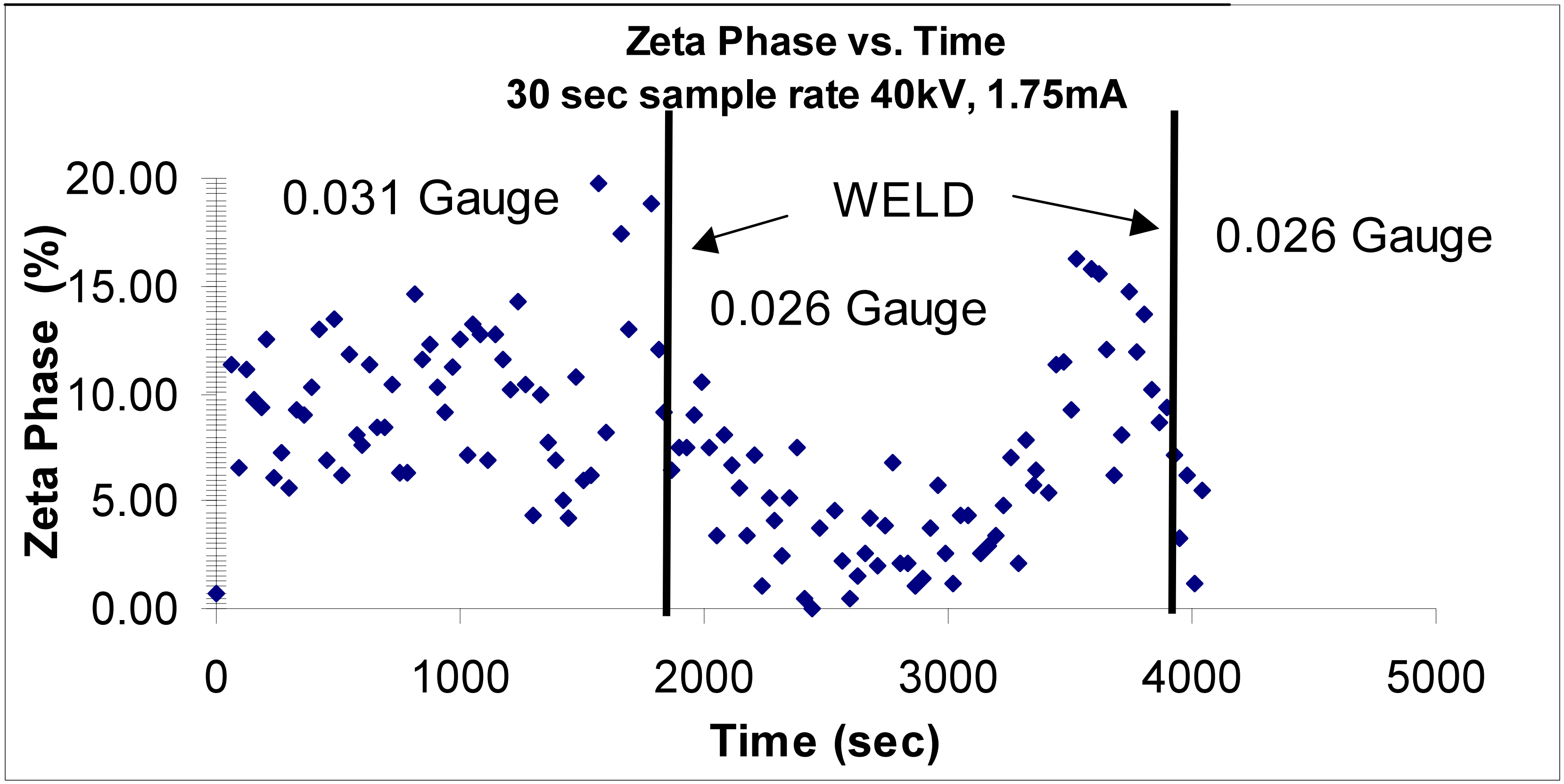
Steel line passing sensor head at between 135 and 320 feet per second

Technical Progress



Final instrument calibration

Technical Progress



Typical on-line analytical results (30 second measurements)

Technical Progress / Market Potential

- **Progress toward milestones/goals**
  - The overall milestones of this project have been clearly met.
  - Samples were taken by AK Steel from the on-line monitored batches and analyzed by conventional means with excellent agreement.
- **Possible barriers**
  - Investment by the industry in alternative solutions over the duration of the project has reduced the near term market for this approach.
- **Market Potential**
  - Confidential discussions are active with a steel manufacturer to adopt this technology in their facilities.

Outlook

- **Industrial end-user involvement**
  - This project has been well supported by AK Steel. Providing access to a galvanneal line whilst not interfering with normal production shows high level commitment and trust.
  - The availability of on-line x-ray optic based measurement technology has opened many other industrial end-user relationships and applications.
  - 2 year development timescales from concept to prototype (longer to system introduction) are unacceptable for many IOF applications.
  - Currently the cost of niche instrument development is high

General Market Potential

- **Other IOF areas of applicability**
  - Texture and alloy phase in aluminum
  - Cement (component phase, free lime content and particle size)
  - Sulfur in fuel (composition)

- **Other industries**
  - Pharmaceutical- active ingredient (quantity and correct polymorph)
  - Semiconductor- thin film crystal orientation, stress and texture
  - HTS texture

Programmatic Merit

- **The XRD System will reduce the energy that is wasted by the production of out-of-spec product:**
  - Galvannealing is the second-most energy-intensive process in steelmaking, second only to ironmaking.
  - Out-of-spec product is sold for applications which would not have required the high energy input.

U.S. and World Energy Savings

	Production (000 N.T.)	Yield Savings (000 N.T.)	Electricity Savings ($ 000)	Other Energy Savings ($ 000)	Total Energy Savings ($ 000)
USA	5,000	50	$521	$509	$1,030
World	18,000	180	$1,876	$1,833	$3,709

Summary

- **Industrial on-line x-ray diffraction measurements have been shown for the first time using x-ray optics.**
- **There is large potential for measurements of this kind, work continues with steel companies to integrate this technology on their production lines.**
- **Development times and costs must be compressed to allow for exploitation of small and large market niches.**
- **The use of modular systems would allow simple development of industrial solutions but requires significant up-front investment.**